Final Report
Non-Destructive Analysis of Hydrogen in Nuclear Power Plant Materials

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Abstract

Researchers at Missouri University's Nuclear Engineering Program and Research Reactor have used a non-destructive, epithermal neutron scattering technique to measure hydrogen in materials of interest. This process utilizes the Hysen or "notched neutron spectrum" technique to measure hydrogen via the moderation of neutrons. The process was first suggested at KAPL and was refined and put into production use for many years at INEL to measure the amount of hydriding in naval reactor fuel. More recently this technique was adapted at the Missouri University Research Reactor (MURR) and was modified to increase its sensitivity by approximately a factor of 20. The resulting system at the MURR has an uncertainty of approximately 0.4 ppm (or 5 micrograms) of hydrogen in steel or other metals of interest. It has been used to measure hydrogen in steel, TiAl, obsidian, silicon, diamond and zirconium.

This report describes a new approach where the notched neutron spectrum is replaced with a monochromator (similar to those used in thermal neutron diffraction studies) to produce a beam of neutrons at a selected energy. For the determination of hydrogen, the energy of the monochromator must typically be in the eV range, allowing the detection of neutrons from hydrogen scattering by resonance absorbers, such as Pu-239 based fission chamber. Pu-239 has a resonance at 0.296 eV with a cross section of 7500 barns, thus implementing on-line measurement of hydrogen at the ppm level.

The MURR has previously developed monochromators using bent crystals of silicon to focus the neutron beam. For this work, we have designed and built a monochromator using a stack of 13 commercial, thin bent crystals of silicon. Calculations indicate a source strength at 1.25 eV of greater than 10^6 n/sec, focused into a spot about 2.5 cm by 2.5 cm.

A computer model was developed for a simplified geometry of the experimental setup using the MCNP code. These results are in good agreement with experimental measured values. The system is capable of a lower limit of detection (LLD) of 8 ppm of hydrogen in steel in a 30 minute counting period under optimu m conditions at the MURR.

Introduction

Due to the ubiquitous nature of water and the fact that water can be ionized into a hydrogen ion and a

hydroxyl radical, hydrogen is literally, universally available. Essentially all metallic surfaces come in contact with hydrogen-containing materials from which the hydrogen can enter the material either as an ion or molecule. Once in contact with the material, because of its small size, the hydrogen can readily diffuse into the substrate materials where it can change the material properties.

Many materials in nuclear power plants are susceptible to hydriding. One material is the zirconium cladding in nuclear fuel pins. As it is corroded by coolant water, a fraction of the hydrogen generated is picked up by the cladding. This hydrogen, either in dissolved form or precipitated as a hydride, results in embrittlement of the cladding. A nondestructive system is needed to detect this hydrogen or hydrides in irradiated fuel cladding. Other materials associated with either nuclear and non-nuclear components will also benefit from this capability.

The technique proposed here uses the elastic scattering of epithermal neutrons from single collisions with hydrogen, and the unique energy-angular relationship of this neutron scattering to achieve high levels of sensitivity.

Technique

Researchers at the University of Missouri-Columbia have used a non-destructive technique (Miller, et al., 1991) to measure hydrogen in materials of interest. This process utilizes the Hysen or "notched neutron spectrum" technique to measure hydrogen via the moderation of neutrons. The process was first suggested at Knolls Atomic Power Laboratory (Gavin, et al., 1969) and was refined and put into production use for many years at Idaho Nuclear Engineering Laboratory (Roger, 1989) to measure the amount of hydriding in naval reactor fuel. More recently this technique was adapted at the Missouri University Research Reactor (MURR) and was modified to increase its sensitivity by approximately a factor of 20 (Miller, et al., 1992). The resulting system at the MURR has an uncertainty of approximately 0.4 ppm (or 5 micrograms) of hydrogen in steel or other metals of interest. It has been used to measure hydrogen in steel (Miller, et al., 1992), TiAl, obsidian, silicon, diamond (Spitsyn, et al., 1993) and zirconium (Buscher, 1994). Typical results are seen in Figure 1. It has also been mo dified for the determination of deuterium (Golshani, 1996) and calculations to model a measurement system for fuel pin analysis (Miller, et al.,

1996) have been performed.

The notched filter technique uses a filtered spectrum of neutrons that has been depleted of neutrons in given energy ranges by absorption at resonance energies by a thick resonance absorber. This spectrum is then passed through the sample to be studied and the resulting neutron flux detected by thin foils using the same resonance absorber. Theoretically, the resulting activation in the foils should be zero unless a sample placed in the notched neutron spectrum slows neutrons down to a resonance energy so they can activate the foil. If the sample material has a relatively large atomic number, i.e., aluminum, iron, zirconium, copper, manganese, or other metal, the amount of slowing down and resulting activation is small. If the sample has hydrogen in it, the slowing down of the neutrons is large and causes activation. Thus, this technique utilizes the appreciable energy loss of neutrons scattered off of hydrogen nuclei.

This measurement technique has been modified (Miller, et al., 1992) by taking advantage of the unique energy/angle relationship of neutrons scattered from single collisions with hydrogen. By carefully selecting the solid angle subtended by the activation foils, the activation due to hydrogen can be increased relative to the activation caused by the host metal. This has increased the sensitivity to hydrogen by approximately a factor of 20 over the original geometry and has lead to sensitivities less than 1 ppm.

The material typically used as the spectrum notching/activation foil is Indium. Indium exhibits several resonances in the 1- to 10-eV range, activates with a 55 minute half-life and emits several, easily detectable gamma-rays. The shortcoming of using Indium is that it takes several hours to saturate the activation foils. Counting must also be done off-line, resulting in several hours to analyze a single sample.

The basic principle that allows the notched neutron spectrum to work is the use of incidence neutrons above the energy of the resonance of the foil/detector. The detection of hydrogen is achieved by single collisions with hydrogen leading to a unique energy/angle relationship for the scattered neutron. By replacing the notched neutron spectrum with a monoenergetic beam of neutrons at a selected energy, other resonance absorbers can be used to detect the scattered neutrons. A quasi-monoenergetic beam can be produced through the use of monochromators similar to those used in neutron diffraction studies and optimized by using bent crystal techniques (Popovici, et al., 1995, 1995a, 1997). The only distinction is that the energy must typically be in the eV range. With this source of neutrons, a resonance absorber such as Pu-239, or other 1/v cross section absorbers such as U-235 or He-3, can be

used as the "detector" and measurements can be implemented on-line.

Monochromator

To extract a monochromatic neutron beam by Bragg reflection from single crystals, the neutron wavelength is given by Eq. (1), expressed through the d-spacing of the crystallographic planes and the Bragg angle 🗎.

(1)

The selected energy band is

(2)

where ❸🗎 represents the angular divergence of the neutron beam. For epithermal neutrons the energy band becomes broad due to the large cot(🗎) factor at shallow Bragg angles.

The efficiency of Bragg reflection is given by the ratio between the integrated reflectivity of the crystal R$_{\theta}$ (θ- scale integration) and the beam angular divergence. With thermal neutrons, the reflection efficiency is increased by using mosaic crystals of large R$_{\theta}$, of which pyrolytic graphite (PG) is the best for high-intensity applications. However, with PG the attenuation of neutrons increases in the epithermal range due to strong multiphonon scattering. In addition, PG is expensive.

The diffracting power of crystallographic planes falls off rapidly with increasing neutron energy. This is partially compensated by the increase of the neutron path in the crystal. However, with PG the path is limited by the strong attenuation in the epithermal range. With silicon, the linear attenuation coefficient is one order of magnitude smaller than for PG and the path stays long. The long path makes bent silicon suitable as a monochromator for short wavelength neutrons (Wagner and Mikula, 1992).

Bent crystal monochromators act as neutron lenses. Diffracted beams can be focused to the sample by a suitable choice of bending radii. With symmetric reflection, the distance to image L_1 and the distance to source L_0 are related through the usual lens Eq. 3:

(3)

where f is the focal length. The focal length has different expressions in the diffraction (horizontal) plane:

(4)

and the vertical plane:

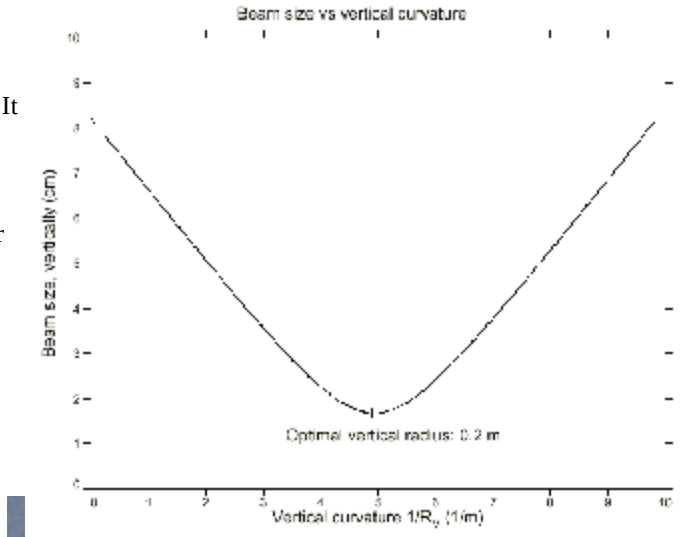
(5)

In Eqs. (4) and (5), R_H and R_V are the corresponding radii of curvature of the crystal. To give the same focal distance at shallow Bragg angles, the radii in the vertical and the horizontal plane must be very different (varying from a fraction of a meter in the vertical plane to tens of meters in the horizontal plane).

At shallow Bragg angles the reflectivity width of bent perfect crystals is just the ratio between the neutron path in the crystal and the radius of curvature in the plane of diffraction. From path considerations, the thickness of silicon monochromators can be around 1 cm. However, at such a thickness silicon cannot be bent because it breaks easily. One can get around the breaking limitation by using bent packets of thin wafers. The reflectivity curve of multilamella packets is generally comb-shaped, with the curves of individual lamellae distanced (distance between teeth larger than tooth width). The filling ratio can be manipulated to optimize the reflectivity (Stoica, et al., in press). With symmetric reflection, the filling ratio approaches unity with decreasing Bragg angle. Multilamella units are thus naturally optimal for epithermal neutrons.

A multilamella unit was previously fabricated for the MURR neutron diffraction stress mapping machine (Witte, et al., 1998). A similar but longer unit, to better cover the beam at shallow Bragg angles, was fabricated for

this project from commercial Si [100] wafers of 12" diameter. It consists of 13 lamellae, each 0.76 mm thick, for an overall thickness of 1 cm. It was optimized for the (400) reflection at the neutron energy of 1.25 eV. (See Figure 2)



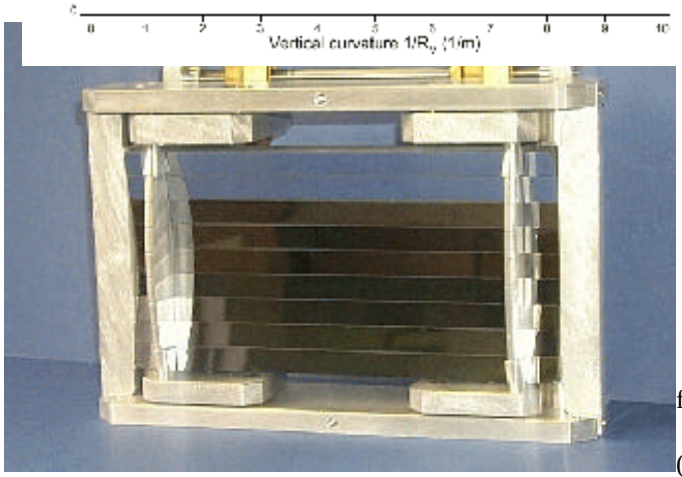


Figure 2 - Monochromator

The DAX code for design of two-axis neutron spectrometers (Popovici, et al., 1995) was used to calculate the beam from this monochromator. The beam sizes at the sample (assuming a source 10 cm diameter at L_0 of ~5 m and a monochromator to sample distance L_1=1.5 m), are shown as a function of the corresponding curvatures in the “z” or vertical axis and the “y” or horizontal axis in Figs. 3 and 4. Expected beam sizes (full widths at half maxima of the intensity distributions) are less than 2 cm in each direction. At a Bragg angle of 5.4° the neutron path in the monochromator exceeds 10 cm. The overall reflectivity width of the assembly is of 0.3° at a peak reflectivity of 7.3%. The selected energy band is 21%. Thus, the neutron intensity through a sample of 2 cm x 2 cm is expected to be around 10^6 neutrons/second.

Figure 3 - Vertical Curvature

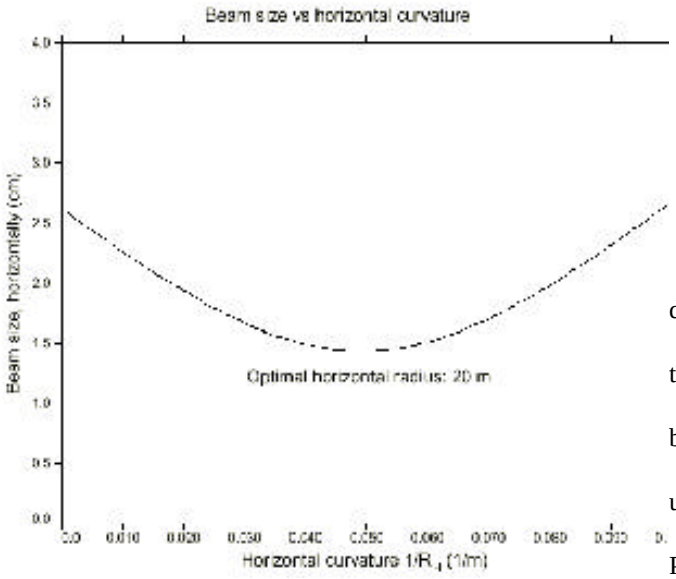


Figure 4 - Horizontal Curvature

Pu-239 Based Detector

Since the neutron detection material is no longer required to notch the spectrum, any material with a resonance below the incident neutron energy can be used to detect neutrons scattered from hydrogen. Pu-239 has been chosen because: 1) it has a resonance at 0.296 eV with a peak fission cross section of 7500 barns and 2) it has essentially a zero background count rate for all radiation events except for neutrons that cause fission in the detector. Fissioning of Pu-239 results in fission products that are produced in the 60 - 100 MeV range which is far greater than other radiation events (either external gamma-rays or internal alpha emissions) that typically deposit a few MeV in the detector. Thus, simple pulse height discrimination can be used to eliminate background radiation.

In the course of this research grant, it was determined that fabricating the plutonium-based detector could

not be accomplished in time for experimental use. Although there are no technical limitations to constructing this detector, problems with licenses (in the commercial sector) and the availability of necessary electroplating equipment (at the national laboratories) made fabrication impossible. Thus, the decision was made to fabricate the fission plate detector out of U-235 instead. U-235 has a cross section similar to Pu-239, although the resonance peak around 0.3 eV is less pronounced. This change in cross section leads to less discrimination between steel and hydrogen and decreases the sensitivity.

In addition to the U-235 based detector, a He-3 gas-filled detector was also added. The advantage of the He-3 detector is a much higher (by roughly two orders of magnitude) sensitivity, which increases the count rate and improves the counting statistics. The disadvantage is an even less favorable cross section shape (which is simply 1/v for this cross section) and a much higher background. With these detectors it was possible to obtain theoretical results for three different detector systems and experimental results for two. In all cases, these detectors could be operated in pulse mode, allowing on-line measurement of hydrogen at the ppm levels in metals.

Monte Carlo Calculations

A Monte Carlo model using MCNP was used to give approximate sensitivities for the three detector systems. The geometry for these calculations assumed the beam of neutrons predicted by the monochromator model, the sample and the detector. As such, it does not include any neutron scattering off of structures and shielding around the experimental area.

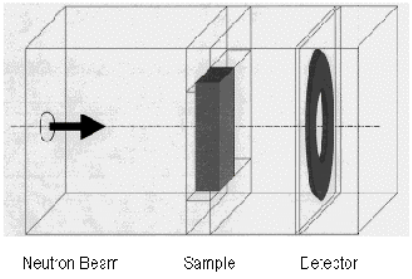
From these calculations it was determined that the optimum incident neutron energy is approximately 1.25 eV for Pu-239 and U-235 interactions. This maximizes the hydrogen signal with respect to the host metal signal. The corresponding angle of placement for the detector is 55 - 75 degrees, which corresponds to plating the fission material onto the circular, flat plate of the detector with a 5 cm inner diameter and 10.8 cm outer diameter placed 2.86 cm from the sample. A thickness of 0.35 microns of fissionable material is plated on two sides of the anode, resulting in a total thickness of 0.7 microns. A thicker layer would not increase the sensitivity due to the limited range of the fission products in this layer.

Figure 5 - Geometry

For the He-3 detector simulation, it was assumed that the detector subtended only a part of the solid angle covered by the fission detector. The results are given in Table I below.

Reactor Implementation

Three different beamtube locations were considered for experimental verification of this technique. One possibility is the "E" beamtube at the MURR. This facility is fitted with a small angle scattering device into which the new monochromator would be inserted. As currently configured, this facility produces approximately 10^{10} n/cm^2/sec thermal neutrons at the monochromator. The "F" beamtube, which directly views the core and would have more neutrons in the epithermal region, may have advantages over the "E" beamtube and was also considered. Finally, beamtube "D" is normally fitted with a prompt gamma neutron activation analysis facility. This facility has the disadvantage that it has only a 2.5 cm diameter beam and has a sapphire filter that allows thermal neutrons to pass but blocks epithermal neutrons (at 1.25 eV it



allows only 4% of the neutrons to penetrate). However, implementation of this research required no modifications of the beamtube area and the hydrogen measurement experiment could be setup in a matter of a few hours. Thus, beamport "D" was used for these measurements in order to measure sensitivities with the understanding that the desired ppm hydrogen sensitivity would necessitate ultimately moving the experimental setup to another beamtube.

Discussion of Results

Computer modeling using the MCNP code was utilized to predict the sensitivity of this method, expressed as counts in the detector per incident neutron per milligram of hydrogen and per milligram of iron. Table I summarizes both the computational results as well as experimental results for an iron sample and a "hydrogen" sample using lucite to simulate the hydrogen content.

Table I - Calculated vs. Experimental Results for Different Detector Systems

	Pu-239		U-235		He-3	
	(Background = 2 c/s) (assumed)		(Background = 2 c/s)		(Background = 58 c/s)	
	Calculated	Exp.	Calculated	Exp.	Calculated	Exp.
Hydrogen (c/n/mg)	1.4×10^{-6}	n.a.	6.0×10^{-7}	3.4×10^{-7}	3.8×10^{-5}	2.8×10^{-5}
Iron (c/n/mg)	1.4×10^{-9}	n.a.	1.8×10^{-9}	3.0×10^{-9}	4.1×10^{-7}	4.8×10^{-7}
Sensitivity Ratio (H/Fe)	1000	n.a.	330	110	90	60
LLD	0.11 mg = 30 ppm		0.31 mg = 90 ppm		0.028 mg = 8 ppm	

The table gives both calculated (using MCNP) and experimental sensitivity as counts in the detector per incident neutron on the sample per milligram of hydrogen or iron in the sample. Multiplying these numbers by the neutron current (i.e. neutrons/sec) incident on the sample gives the counting rate in the detector.

The calculated sensitivity is similar for the Pu-239 and U-235 detectors and much larger for the He-3 detector. This is because the thickness of fissionable material in the detector must be limited. It is also seen that the

He-3 has the largest background for the experimental measurements due to the same phenomenon.

The ratio of sensitivities between hydrogen and iron is given in the next to last row. As expected the greatest difference is for the Pu-239 detector, less for the U-235 and even less for the He-3 which indicates that the Pu-239 would have a lower background count rate from the host material containing the hydrogen.

Experimental results for the U-235 and He-3 detectors are also given in the table to compare with Monte Carlo calculated results. The He-3 detector was used to estimate a flux of 2000 n/sec on the sample, which agreed exactly with the prediction from the monochromator model. With this normalization, there is general agreement between experiment and calculation. The experimental hydrogen sensitivities are consistently less and the iron sensitivities are consistently more than calculated, which also results in a lower hydrogen to iron sensitivity ratio. This is because the Monte Carlo geometry does not include neutrons scattered off of the shielding materials around the sample position.

Finally, these sensitivities were combined with the flux that could be achieved if the experiment were moved to beamport "E" or "F" which would increase the neutron flux on the sample from 2000 to approximately 10^6 n/sec. The corresponding lower limits of detection (LLD) were calculated, assuming that the background count rate in the Pu-239 detector would be the same as in the U-235 detector. This resulted in the lowest lower limit of detection (LLD) for the He-3 detector of 8 ppm.

Although the He-3 has the lowest sensitivity ratio between hydrogen and iron, it has a smaller background to signal ratio. In the current setup the background from scattered neutrons in the experimental area is far greater than the background from the iron in the sample, thus the sensitivity ratio is not critical. An optimized shielding arrangement which would reduce the "area" background might change this relative importance and one of the other detectors might become optimum.

Conclusions

This technique is capable of measuring hydrogen in nuclear material systems, including those that may be radioactive. This is an advantage since other nuclear techniques for measuring hydrogen (such as prompt gamma neutron activation analysis) are sensitive to radioactive background. The technique is also applicable to any other industrial material requiring non-destructive analysis of hydrogen content.

Since the integrating foil detector has been replaced with an active detector, the measurements can be made on-line. Previous implementation of the notched neutron spectrum technique required several hours to activate the foil and then several hours to measure the result. With on-line measurement, the time necessary to analyze a sample will be shorter and sample throughput will be increased.

The measurement technique will require a source of neutrons, a monochromator and a thermal neutron detector. Samples (from nuclear systems or from any industrial application) that need to be analyzed are simply placed in a sample holder and the hydrogen content is deduced from the detector count rate. The beamtube of a research reactor is optimum, but collimated beams from accelerator based neutron sources are envisioned.

For the specific application to nuclear fuel pin hydriding, this technique may ultimately be adaptable for in-pool or near-pool analysis of nuclear fuel pins. This application would require an accelerator-based neutron source. An appropriate geometry utilizing the accelerator, a monochromator, the fuel pin and the detector system would yield a hydrogen concentration. This would eliminate the need for irradiated-fuel transport from the power plant and expensive hot-cell inspections.

Publications

Two publications have resulted from this work to date:

Miller, W. H., M. Popovici, and L. Groza, "Non-Destructive Analysis of Hydrogen in Nuclear Power Plant Materials," Eighth International Conference on Nuclear Engineering (ICONE 8), American Society of Mechanical Engineers (ASME), Baltimore, Maryland (April 2-6, 2000).

Miller, W. H., M. P. Popovici, and R. L. Groza, " Experimental Verification of Epithermal Neutron Scattering for Determining PPM Hydrogen in Metals," Transactions of the American Nuclear Society (submitted for publication)

Future Work

Although contractual commitments to DOE have been completed, graduate student R. L. Groza has not completed his Ph.D. dissertation related to this research. Additions to the Monte Carlo simulation are needed to include more of the shielding in the experimental area. This model can then be used to refine the shielding geometry to reduce the background which is the current limit to sensitivity. More work with the He-3 detectors is needed

since the current design does not make use of the entire solid angle into which hydrogen neutrons are scattered. The 1.25 eV energy beam was also chosen as optimum for the Pu-239 detector, and may not be optimum for either the U-235 or H-3 detectors. A pyrolytic graphite monochromator is available, is more efficient at lower neutron energies, and could be used to compare with the results from the silicon monochromator. These, and other variables, will be evaluated. We anticipate that additional publications will be forthcoming with the completion of this work and Mr. Liviu's dissertation.

Acknowledgments

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